MISSOURI DEPARTMENT OF NATURAL RESOURCES
AIR AND LAND PROTECTION DIVISION
ENVIRONMENTAL SERVICES PROGRAM
Standard Operating Procedures

SOP #: MDNR-FSS-006B EFFECTIVE DATE: March 21, 2002

SOP TITLE: Sampling Soils and Other Solid Media for Volatile Organic Analysis (VOA)

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SUMMARY OF REVISIONS: Minor updates were made to the 1999 version of this SOP.

APPLICABILITY: The procedures described in this SOP are applicable to all ESP personnel who collect soils or other solid media samples for VOA.

DISTRIBUTION: MoDNR Intranet
ESP FSS Section Chief
ESP SOP Coordinator

RECERTIFICATION RECORD:

Date Reviewed				
Initials				

1.0 SCOPE AND APPLICABILITY

This Standard Operating Procedure (SOP) describes the sampling methodology to be followed by Environmental Services Program (ESP) personnel when collecting soil or other solid media samples for volatile organic analysis (VOA). This SOP does not apply to water or other liquid media. Refer to MDNR-FSS-006A *Sampling Water and Other Liquids for Volatile Organic Analysis (VOA)* for guidance in collecting water and other liquid samples for VOA.

2.0 SUMMARY OF METHOD

Volatile organic compounds are the most sensitive group of chemicals that are routinely analyzed in environmental investigations. Aeration, agitation, heat, pressure changes, and microbial degradation reduce the amount of volatiles in a sample and can result in data that are negatively biased. Loss of volatiles through poor sampling techniques and microbial degradation can be reduced by minimizing sample disturbance, eliminating headspace in sample containers, and properly preserving samples. This SOP describes the methodology that should be followed when collecting soil and other solid media samples for VOA. Procedures for preservation techniques, sample handling, and transportation are also covered. By following the methods described in this SOP, sampling personnel will be able to collect volatile organic samples that are representative of the media sampled, thus providing valid data.

3.0 DEFINITIONS AND ACRONYMS

- En Core Sampler - Disposable, single-use sampling device used to collect and contain a soil sample for VOA
- HASP - Health and Safety Plan
- OSHA - U.S. Occupational Safety and Health Administration
- PID - Photoionization Detector - Portable air monitoring instrument used to measure the amount of ionizable organic vapors present
- TCLP - Toxicity Characteristic Leaching Procedure
- Volatile Organics - Organic compounds that have a boiling point less than 200 °C

4.0 HEALTH AND SAFETY REQUIREMENTS

4.1 Sampling personnel need to be aware of the hazards of working with methanol, the preservative that may be used when collecting soil samples for VOA. Field preservation with methanol is not always necessary (see Section 8.0). However, if field preservation with methanol is needed, personnel should be aware that methanol is a highly flammable liquid with a flash point of 12 °C (54 °F) and should exercise caution when working near potential sources of ignition. Methanol is also a poison and should be handled with care to avoid skin contact, breathing vapors, and ingestion. All sampling personnel should wear safety glasses when using methanol. Material Safety

Data Sheets are available to all ESP personnel who want further information on methanol.

4.2 For sampling investigations that occur at hazardous waste sites, more detailed health and safety requirements will be described in site specific HASPs. Personnel who conduct work at hazardous waste sites will be expected to read and comply with the requirements of any site specific HASP.

4.3 All ESP personnel who conduct work at hazardous waste sites will be required to participate in the department's medical monitoring program in accordance with Divisional policy.

5.0 PERSONNEL QUALIFICATIONS

5.1 Hazardous Waste Operations and Emergency Response (HAZWOPER) Site Work

All ESP personnel directly involved in field investigations at sites that fall under the EPA Worker Protection requirements of 40 CFR Part 311, referencing OSHA 29 CFR Part 1910.120, and meet the definition of HAZWOPER activities must meet the following qualifications:

- Attend a 40-hour course designed to meet the OSHA health and safety training requirements for hazardous site workers;
- Attend an annual 8-hour health and safety refresher course, or receive equivalent training;
- Participate in a medical monitoring program in accordance with Divisional policy;
- Receive appropriate on-the-job training;
- Be familiar with the *Hazardous Substance Emergency Response Plan*, written and maintained by the ESP;
- Be familiar with the ESP SOP manual and have read all SOP documents that are applicable to the field activities, including but not limited to those referenced in this SOP.

5.2 Non-Hazardous Site Work

All ESP personnel who conduct field or sampling investigations at sites that do not fall under HAZWOPER regulations must meet the following qualifications:

- Receive appropriate on-the-job training;
- Be familiar with the ESP SOP manual and have read all SOP documents that are applicable to the field activities, including but not limited to those referenced in this SOP.

6.0 SUPPLIES AND EQUIPMENT

6.1 At a minimum, the following supplies and equipment will be needed to properly collect, preserve, and handle soil or other solid media samples for VOA:

- 5-gm En Core Sampler devices
- En Core T-handle
- 2-ounce glass containers with Teflon-coated lid liners
- nitrile gloves
- plastic ziplock bags
- cooler with ice
- sample labels (both pre-numbered and blank)
- Field Sheet and Chain-of-Custody Record forms
- field notebook
- level D personal protective equipment

6.2 Other equipment and supplies may also be needed depending upon special requirements that may be specified in a sampling plan and/or a HASP, the type of media sampled, or other site-specific circumstances. The following list is not intended to be all-inclusive but merely suggests many common optional supplies and equipment that may be helpful when conducting a field investigation that involves the collection of soil or other solid samples for VOA:

- PID
- field decontamination supplies
- shovel
- trowels or spoons
- camera
- level B or C personal protective equipment
- bottle of analyte-free water for the collection of rinsate blanks and/or field blanks
- analytical balance
- pre-preserved, pre-labeled, pre-weighed 40 ml vials

7.0 PROCEDURE

7.1 Collection of Soil and Sediment Samples

The En Core Sampler device is appropriate for use in most soils, sediments, and other finer-grained material that can be cored into with the hard plastic En Core Sampler device. Materials that are too wet or too rocky or otherwise cannot be collected using an En Core Sampler should instead be collected in a 2-ounce glass jar with a Teflon-lined lid using a clean stainless steel spoon (refer to Section 7.2).

7.1.1 Determine whether or not TCLP analysis may be needed. If the only analysis needed is total VOA, then two 5-gram En Core Sampler devices will need to be filled per sample. If TCLP analysis is needed, then four 5-gram En Core Sampler devices will be needed per sample.

7.1.2 Select an En Core Sampler device and tear open the top of the bag. Note that the bag has a ziplock feature at the top. Do not destroy the bag, as it will be reused for sample storage after the sample has been collected. Wear a pair of new nitrile gloves whenever handling the sampling equipment.

7.1.3 Push the plunger rod down on the En Core Sampler device and attach to the T-handle. Ensure that the En Core Sampler device is locked into place. Refer to Appendix A which is a copy of the manufacturer's step-by-step instructions, including diagrams, for using the En Core Sampler device.

7.1.4 Remove the cap on the En Core Sampler device. The cap must be kept clean since it will be used to cap the device once a sample has been collected. Using the T-handle, push the En Core Sampler device into the soil, effectively taking a core sample of the soil. Push until the device is completely full. The objective is to fill the device so there is no headspace or voids present. Remove the sampler from the soil and quickly wipe off any excess soil from the outside of the device using clean, dry paper towels. Immediately cap the En Core Sampler device with the locking cap.

7.1.5 Place the En Core Sampler device back into its original bag. Affix an ESP sample label to the outside of the bag. Ensure the ziplock seal on the bag is closed and put the bag with the sample inside into a cooler on ice.

7.1.6 Refer to MDNR-FSS-003 *Sample Numbering and Labeling* for further guidance on using sample labels.

7.2 Collection of Other Solid Media (non-soils)

Solid materials that are either too wet or too coarse-grained to be collected with an En Core Sampler should be collected in a wide-mouth glass jar with a Teflon-lined lid.

7.2.1 Use new, certified-clean, 2-ounce glass jars with Teflon-lined lids for each sample. Two 2-ounce glass jars should be sufficient for analyses of both total VOA and TCLP VOA, if necessary.

7.2.2 Use a new stainless steel spoon to scoop the sampled material into the glass jars.

7.2.3 Fill each jar as full as possible. Pack the material tight using the back of the spoon in an attempt to eliminate any headspace in the jar. If the threads of the

jar got dirty during sample collection, wipe the threads clean with paper towels before sealing with the lid to help ensure a good seal.

7.2.4 Samples collected for VOA should be transferred directly from the source to the sample container and should never be mixed or homogenized. Mixing a sample in a pan or other device agitates and aerates a sample and will result in the loss of volatiles. Therefore, the collection of replicate split samples of a solid media for VOA is not recommended.

7.2.5 Samples must be labeled and preserved on ice as soon as possible after collection. Refer to MDNR-FSS-003 *Sample Numbering and Labeling* for procedures on using sample labels.

7.3 General Considerations

7.3.1 At a minimum, always wear a new pair of clean nitrile gloves for each sample collected. Using a clean pair of nitrile gloves for each sample serves a dual purpose: personal protection and reducing the potential for cross-contamination among samples.

7.3.2 Regardless of the type of media sampled, attempt to collect samples in the order of least contaminated to most contaminated. This is especially important if any of the sampling equipment is non-dedicated and will be reused while at the site. Refer to Section 9.5 for collection of rinsate blanks if non-dedicated equipment is used.

7.3.3 Whenever processing samples near vehicles, such as is commonly done on the tailgate of a truck, always shut off the engine to eliminate any potential effects on the samples from the engine exhaust.

7.3.4 Use a bound field notebook to record pertinent information when conducting any type of field investigation. Refer to MDNR-FSS-004 *Field Documentation* for guidance in recording permanent notes during a field investigation.

7.3.5 In addition to using a field notebook for documentation, all samples that are returned to the ESP laboratory for analysis or evidentiary purposes must be recorded on a Field Sheet and Chain-of-Custody Record. Refer to MDNR-FSS-002 *Field Sheet and Chain-of-Custody Record* for guidance in using this important documentation record.

8.0 HANDLING AND PRESERVATION

8.1 Regardless of whether a sample has been collected in an En Core Sampler device or a glass sample jar, all samples should be chilled immediately upon collection to

approximately 4 °C. This is typically accomplished by placing the samples on ice in a cooler. If it is necessary to keep the samples on ice for extended periods of time, the coolers should be periodically drained to ensure that the samples do not become submerged in water from melted ice. In addition to keeping coolers drained, placing the sample containers in ziplock bags helps keep sample containers dry.

8.2 The holding time for a sample collected in an En Core Sampler device is 48 hours. Within 48 hours of collection, the En Core sample must either be frozen (which extends the holding time to 14 days) or extracted using appropriate solvent. Due to the relatively short holding time, it is extremely important that the sample collector provide prior notification to the CAS regarding the collection activities. There is a freezer located in the ESP decon room that should be used for overnight storage of soil samples that cannot be turned in to the CAS within 48 hours.

8.3 If the samples cannot be returned to the ESP within 48 hours to be either frozen or extracted, then the samples must be preserved in the field with either methanol or water. The preservation method will be dependent upon the data needs and the suspected level of contamination. Samples that are suspected to be contaminated with volatile organics at levels less than 200 ppb should be preserved with water. Samples that are expected to have a higher level of contamination should be preserved with methanol. The sample collector should be aware that preservation with methanol results in a higher detection limit than that obtained using water as a preservative.

8.3.1 Field preservation with either methanol or water requires 40-ml VOA vials that are pre-weighed in the lab with 10 mls of the appropriate extraction solvent (methanol or water) added prior to being pre-weighed. Sample labels must also be affixed to the sample vials prior to being pre-weighed since the level of precision required is 0.01 gram.

8.3.2 The sample is first collected using the En Core Sampler device following steps 7.1.1 through 7.1.4.

8.3.3 Once the sample is collected, the soil core is then extruded into a pre-weighed VOA vial. The vial is then capped and weighed in the field on a balance that has the capability to weigh the sample to the nearest 0.01 gm. The balance must be calibrated in the field prior to weighing any samples. Record the total weight of the vial that includes the cap, the soil sample plug, the preservation solvent, and the sample label.

8.3.4 Place the vial into a ziplock bag and place the sample into a cooler on ice.

8.4 The holding time for a sample collected in a glass jar with a Teflon-lined lid is 14 days for volatile organic parameters. There is no chemical preservative used.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

The type and frequency of field QA/QC samples needed at any given site are generally covered by Quality Assurance Project Plans. The different types of field QA/QC samples are listed below, with general recommendations on when they may be appropriate. Refer also to MDNR-FSS-210 *Quality Assurance/Quality Control for Environmental Data Collection* for further guidance and information on QA/QC sampling procedures.

9.1 Trip Blank - Soil

9.1.1 Trip blanks are generally not collected when only soil or other solid media samples have been collected for VOA. A soil trip blank usually consists of a clean sand or other natural soil material that has been baked to ensure it is free of any volatile organics.

9.1.2 If a trip blank is requested for a particular site, contact the CAS at least a week prior to conducting field work in order to obtain a trip blank for solid media. Preparation of a solid media trip blank is much more involved than the preparation of an aqueous trip blank.

9.2 Trip Blank – Preservative

Whenever soil samples must be preserved in the field with either methanol or water, a trip blank of the preservative should be collected. A preservative trip blank consists of an unopened pre-preserved vial that contains 5 ml of the same type of preservative that the soil samples are preserved with. A preservative trip blank will be used to evaluate contamination error associated with sample handling or shipment, or lab handling and analysis.

9.3 Field Blank - Soil

Like trip blanks, collection of a field blank for solid media is rare. Preparation of a field soil blank would be the same as that of a trip soil blank. However, handling would be different. A trip blank is never opened in the field. A field blank is opened while in the field for a short period of time (5-15 minutes) to help determine if there are any air contaminants, such as dust or volatile organic emissions, that may affect a sample as it is collected.

9.4 Field Blank – Preservative

Whenever soil samples must be preserved in the field with either methanol or water, a field blank of the preservative should also be collected. A preservative field blank consists of a pre-preserved vial that contains 5 ml of the same type of preservative that is used to preserve the soil samples. The field blank is opened for a short period of time, approximately the same amount of time a soil sample vial is opened. A

preservative field blank is collected to ensure that nothing from the environment (essentially air contaminants) is contributing to contamination of a sample.

9.5 Rinsate Blank

A rinsate blank may be collected any time non-dedicated sampling equipment is used to obtain more than one sample for volatile organics. Non-dedicated sampling equipment requires decontamination between samples and must be periodically tested to ensure that the equipment is not contributing to cross contamination. Rinsate blanks are collected by pouring analyte-free water over decontaminated sampling equipment to test for residual contamination. The blank water is collected in VOA vials that have been pre-preserved with hydrochloric acid. The blank sample is then submitted for analysis of volatile organics.

9.6 Duplicate

A duplicate is a sample obtained from the same location at essentially the same time as the true sample. The procedures used to collect both the true sample and the duplicate sample should be identical. The true sample and the duplicate sample are analyzed for the same set of parameters. A duplicate soil sample is used primarily to determine the variability or heterogeneity of the matrix. Unlike water, which is a relatively homogeneous matrix, soil is heterogeneous. Due to the heterogeneity of soils, caution must be used if attempting to assess precision associated with sampling methodology or analytical procedures with results obtained from soil duplicate samples.

9.7 Background

Background samples are collected from locations either on- or off-site where little or no contamination is expected to exist. Background samples are collected in an attempt to determine the natural composition of the soil matrix. This is especially important when metals are constituents of concern at a site since metals are naturally-occurring elements. Volatile organic compounds are not expected to be present in natural soils. However, background samples for VOA may be collected if there are off-site sources of contaminants suspected in addition to those sources known or suspected to exist on-site and a distinction must be made between the two.

10.0 REFERENCES

MDNR Environmental Services Program, MDNR-FSS-001 *Required/Recommended Containers, Volumes, Preservatives, Holding Times, and Special Considerations*

MDNR Environmental Services Program, MDNR-FSS-002 *Field Sheet and Chain-of-Custody Record*

MDNR Environmental Services Program, MDNR-FSS-003 *Sample Numbering and Labeling*

MDNR Environmental Services Program, MDNR-FSS-004 *Field Documentation*

MDNR Environmental Services Program, MDNR-FSS-006A *Sampling Water and Other Liquids for Volatile Organic Analysis (VOA)*

MDNR Environmental Services Program, MDNR-FSS-210 *Quality Assurance/Quality Control for Environmental Data Collection*

APPENDIX A

En Core Sampler
Manufacturer's Instruction Page

Disposable En Core™ Sampler

SAMPLING PROCEDURES

USING THE En Core™ T-HANDLE

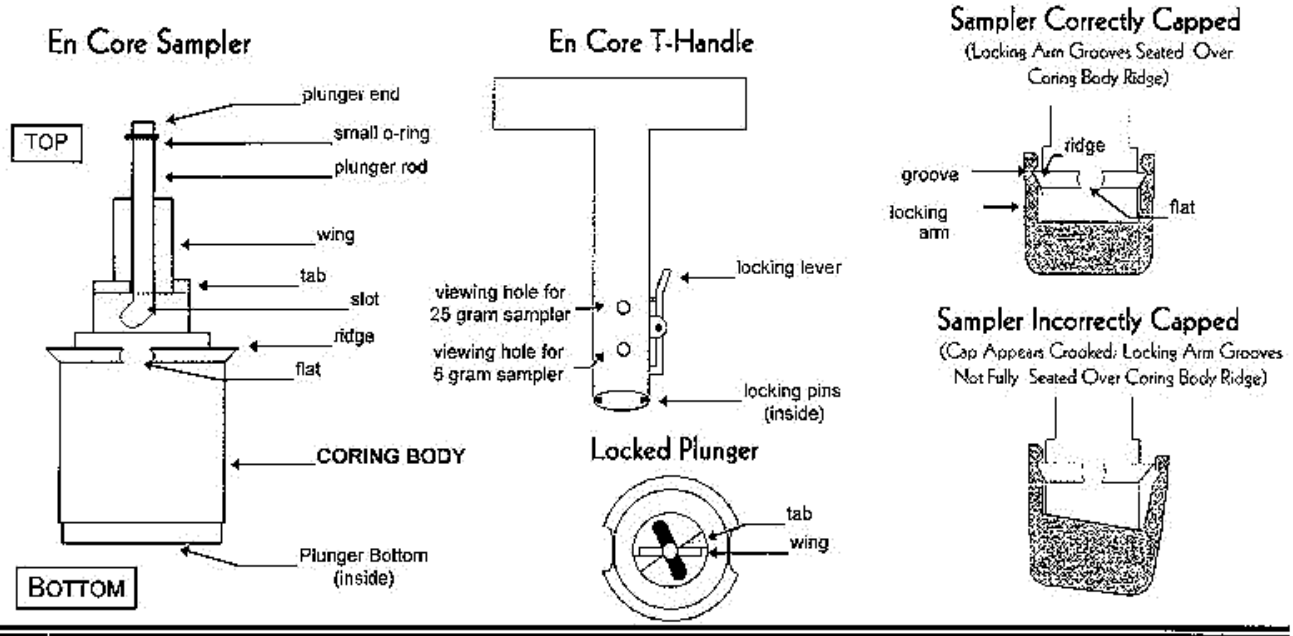
En Novative Technologies, Inc.
1241 Bellevue Street
Green Bay, WI 54302
Telephone: 920-465-3960 • Toll Free: 1-888-411-0757
Fax: 920-465-3963

NOTE:

1. En Core Sampler is a SINGLE USE device. It cannot be cleaned and/or reused.

2. En Core Sampler is designed to store soil. Do not use En Core Sampler to store solvent or free product!

3. En Core Sampler must be used with En Core™ T-Handle and/or En Core™ Extrusion Tool exclusively. (These items are sold separately.)



BEFORE TAKING SAMPLE:

1. **Hold coring body** and push **plunger rod** down until **small o-ring** rests against **tabs**. This will assure that plunger moves freely.

2. Depress **locking lever** on En Core T-Handle. Place coring body, **plunger end** first, into open end of **T-Handle**, *aligning the (2) slots on the coring body with the (2) locking pins in the T-Handle.* Twist coring body clockwise to lock pins in slots. Check to ensure Sampler is locked in place. Sampler is ready for use.

TAKING SAMPLE:

3. Turn **T-Handle** with T-up and coring body down. This positions plunger bottom **flush** with **bottom of** coring body (ensure that plunger **bottom is in position**). Using T-Handle, push Sampler into soil until coring body is completely full. When full, small o-ring will be centered in **T-Handle viewing hole**. Remove Sampler from soil. Wipe excess soil from coring body exterior.

4. Cap coring body while it is still on **T-handle**. *Push* **cap** over **flat** area of **ridge** *and twist* to lock **cap** in place. **CAP MUST BE SEATED TO SEAL SAMPLER** (see diagram).

PREPARING SAMPLER FOR SHIPMENT:

5. Remove the capped Sampler by depressing locking lever on **T-Handle** while twisting and pulling Sampler from T-Handle.

6. Lock plunger by rotating extended plunger rod fully counter-clockwise until **wings** rest firmly against tabs (see plunger diagram).

7. Attach completed circular label (from En Core Sampler bag) to cap on coring body.

8. Return full En Core Sampler to zipper bag. Seal bag and put on ice.